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Bell Laboratory Report 62-21(C) Rev A 3 April 1963

INORGANIC CHEMISTRY
PROCESS IMPROVEMENT

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INORGANIC CHEMISTRY PROCESS IMPROVEMENT

Bell Laboratory Report
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ABSTRACT

- 1. Significant progress was made in the direct deposition of nickel on 6061 aluminum pretreated by two different methods.
- 2. Barrett sulfamate nickel plating baths were put into operation for use in electroforming.
- 3. The Dalic Plating Process was installed and plating techniques developed.

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I. SUMMARY

The following is a resume of the past year's R&D effort in the field of Inorganic Chemistry.

- (1) Significant progress was made in the direct deposition of nickel on 6061 aluminum pretreated by two different methods, neither of which involves the preliminary zincating and electrolytic copper deposition.
- (2) A seven-gallon Barrett sulfamate nickel plating bath has been put into operation for use in electroforming.
- (3) The above mentioned sulfamate nickel plating bath is in operation in one of six temperature controlled, fume-exhausted processing tanks installed this year, with a supporting 10.5 foot x 3 foot drain/spill trough for use in metals cleaning, pickling, and plating investigations.
- (4) The Dalic Plating Process was installed and plating techniques developed.

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II. INTRODUCTION

This report covers Bell Aerosystems Company sponsored research and investigations in the field of Inorganic Chemistry. The general area of interest and application is process improvement.

In order to improve existing manufacturing processes and/or develop new ones, company funds were expended in the installation of new facilities for the study of metals cleaning, pickling and plating. Specific studies were made on protective coatings and/or improvement of component part fabrication.

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III. BACKGROUND

The new materials necessary for Aerospace environments will present problems which electrodeposition may solve. The demand for close tolerances and fabrication of parts that cannot be made by other means creates a demand for processing capability. The success of electroforming nickel on an aluminum mandrel, then dissolving the mandrel with caustic has previously been reported. Nickel can be plated from a variety of baths. The resultant deposit characteristics, such as hardness, ductility, and grain structure, are related to the bath composition. The sulfamate nickel plating process was evaluated for its use in electroforming. The process gives a very high rate of nickel deposition with minimum difficulty and will be used in all future efforts.

Repair of parts with damaged electrodeposited protective coatings, reclamation of worn or mis-machined parts, plating of parts with difficult geometry (deep recesses, etc.), and on the job plating without immersion of the part presented a need for a process for the selective deposition of coatings. A process with these capabilities could save time and money. Dalic Plating, a process of selective plating using specially compounded metallorganic electrolytes held in absorbent materials attached to portable electrodes, was evaluated and is now operational.

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The Dalic process has been tried successfully, on a test specimen basis, for the electrodeposition of nickel and cobalt on aluminum, rhodium and chromium on brass, and palladium on tantalum.

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IV. PROCEDURE

A. ELECTROPLATING PROCEDURES

1. Electroforming with Nickel

A 6061-775 aluminum mandrel for use in electroforming small thrust chambers was turned in a lathe from 3/8 inch round stock to a constriction of 1/16 inch throat diameter. It was prepared for the flash plating of nickel by the Dalic Process by degreasing, chucking in the turning head and rotating slowly, electrocleaning using Dalic cleaning and deoxidizing solution and 10 to 15 volts forward current (mandrel is cathode), rinsing with water, etching using Dalic No. 2 etching solution and 12 to 18 volts reverse current, and rinsing again.

After a flash coat of about 2.5 mil thickness of Epais nickel was deposited on the turning mandrel using 12 to 16 volts, it was rinsed and without drying, was chucked in a stirring motor and immersed in a Barrett sulfamate nickel bath where, at 135°F, 6-8 volts and 2-5 amps, the deposition of nickel was continued at about,0.1 mil per minute until a coating of 0.023 to 0.050 inch was built up (7 hours).

The ends were then cut off, and the aluminum mandrel dissolved out using a sodium hydroxide, sodium gluconate solution.

B. DALIC PLATING PROCESS ON MOTOR MOUNT LUGS

The equipment necessary for the selective plating is transportable. It was moved to the department and the lugs were processed as follows:

- (1) Degrease with methylene chloride (or a suitable solvent)
- (2) Electroclean using Dalic cleaning and deoxidizer solution at 10 volts, forward current, until the following rinse water does not break on the surface. Keep tool in constant motion.

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- (3) Rinse thoroughly with water.
- (4) Etch using Dalic No. 2 etching solution at 8 volts, reverse current until a uniform surface is obtained. Keep tool in constant motion.
- (5) Water rinse
- (6) Etch using Dalic No. 3 etching solution at 15 volts reverse current, until the surface will not become any lighter in color.
- (7) Rinse thoroughly with water.
- (8) Dalic electroplate with Cadmium Acid Code 2020 to the desired thickness.
- (9) Rinse thoroughly with water and dry.

Note: The above operations are to be performed rapidly in the sequence stated and the plating tool must be in constant motion to avoid "burning" the plate. All the solutions used were caught in a catch basin. The solutions are nontoxic so no harmful effect can be expected from the intermixing of liquids.

Facilities and Equipment

The layout of an electroplating setup on a laboratory basis has been completed and has resulted in increased capabilities. The layout and setup for the Dalic process has been completed.

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V. RESULTS

As a part of the continuing investigation of the electroforming of small thrust chambers of the type required for reaction control systems, nickel has been directly plated on 6061 aluminum by two different methods. The Dalic technique, already described in Section IV, can be applied to selected areas of aluminum parts, as a protective coating, or as a base on which to deposit other coatings by electrolytic or electroless means. The other method, phosphoric acid anodizing, since it involves immersion of the entire part, would require masking for application to selected areas. However, this may be used as a suitable base for all electrodeposited coatings on all commercial aluminum alloys except those containing appreciable amounts of magnesium.

A seven-gallon Barrett sulfamate nickel plating bath has been put into operation and is being used to attempt the electroforming of thrust chamber units with throat diameters of less than 0.25 inches. The Barrett process is a method of electrodepositing nickel having exceptionally low tensile stress without the use of addition agents. The bath is a concentrated solution of pure nickel sulfamate $\begin{bmatrix} \text{Ni}(\text{SO}_3\text{NH}_2)_2 \end{bmatrix}$ buffered with boric acid permitting the use of high current densities at lower operating temperatures. It deposits low stressed nickel of high chemical purity with excellent grain structure and ductility.

This sulfamate nickel plating bath is in operation in one of six temperature controlled, fume-exhausted processing tanks installed this year with supporting 10.5 foot by 3 foot drain/spill trough for use in metals cleaning, pickling and electrolytic and/or electroless plating investigations. Both stainless stell, and Koroseal lined seven-gallon tanks were fabricated, and both stainless steel and silica jacketed heating elements procurred, to make

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this experimental processing setup as flexible as possible. A heavy duty Hobart d-c generator of sufficient output for any presently contemplated investigation completes the installation.

The Dalic plating process installation, also new this year, has already been put to profitable use in the replating of the gimbal lugs of nine Agena engine motor mounts, four of which were done in the shop, without dismantling any of the assembled components. The parts had previously been plated with cadmium but were oversize, causing basis metal exposure when machined to size. The Dalic plating recoated the basis metal successfully with a known thickness of cadmium. This salvage technique saved the company considerable money with no disruption of production schedules.

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VI. CONCLUSION

The chemical and electro processing setup and the Dalic plating installation acquired this year furnish a flexible capability for investigation into many surface finishes and electrochemical techniques including electroforming. Some of these are presently under investigation and others will be approached this coming year.

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